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Thermophysical Properties of IoNanofluids Composed of 1-ethyl-3-methylimidazolium Thiocyanate and Carboxyl-functionalized Long Multi-walled Carbon Nanotubes

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Abstract: The concept of IoNanofluids (INFs) as the stable dispersions of nanoparticles in ionic liquids was proposed in 2009 by Nieto de Castro's group. INFs characterize exciting properties such as improved thermal conductivity, non-volatility, and non-flammability. This work is a continuation of our studies on the morphology and physicochemistry of carbon-based nanomaterials affecting thermal conductivity, viscosity, and density of INFs. We focus on the characterization of dispersions composed of long carboxylic group-functionalized multi-walled carbon nanotubes and 1-ethyl-3-methylimidazolium thiocyanate. The thermal conductivity of INFs was measured using KD2 Pro Thermal Properties Analyzer (Decagon Devices Inc., Pullman, WA, USA). The viscosity was investigated using rotary viscometer LV DV-II+Pro (Brookfield Engineering, Middleboro, MA, USA). The density of INFs was measured using a vibrating tube densimeter Anton Paar DMA 5000 (Graz, Austria). The maximum thermal conductivity enhancement of 22% was observed for INF composed of 1 wt% long carboxylic group-functionalized multi-walled carbon nanotubes.

Keywords: IoNanofluids; thermal conductivity; viscosity; density

1. Introduction

Nanofluids consisting of ionic liquids (ILs) and nanoparticles, the so-called IoNanofluids (INFs), have recently attracted attention due to their thermal properties. It is reported that the modification of ILs by dispersing carbon nanoparticles—as one of the most promising macromolecular nanoarchitectures—leads to the improvement of thermal properties [1–6]. Loading of multi-walled carbon nanotubes (MWCNTs) in ILs leads to the enhancement in the thermal conductivity of INFs due to the high thermal conductivity of MWCNTs [2]. Such an amalgamation makes INFs especially attractive as potential heat transfer media which could be beneficial to modern industry. Recently, we found that loading of 1 wt% MWCNTs with length-to-diameter ratio (aspect ratio) of 6300 and

11,000 in 1-ethyl-3-methylimidazolium thiocyanate ([C_2C_1im][SCN]) leads to thermal conductivity enhancement of 43.1% and 43.9% at 25 °C, respectively [5]. Therefore, in this work, we examined the effect of carboxylic group-functionalization of MWCNTs, with an aspect ratio of 10,000, on the thermal properties of cyano-based INFs. To the best of our knowledge, the thermal conductivity of INFs containing carboxylic group-functionalized MWCNTs (oMWCNTs) has never been investigated. Moreover, for the potential application of INFs as heat transfer media, a compromise between heat transfer capability and rheological characteristics is required [7]. Interestingly, the addition of 0.1 wt% oMWCNTs to 1-propyl-3-methylimidazolium iodide with an aspect ratio of ca. 5000, which is extensively used in dye-sensitized solar cells, reduces the viscosity by 71.0% at 25 °C [8]. Similarly, the addition of 0.1 wt% MWCNTs with an aspect ratio of ca. 250 into trihexyltetradecylphosphonium phosphinate substantially reduces the viscosity by 82.4% at 25 °C [7]. On the other hand, thermal conductivity of this INF increases negligibly by 1.5% at 25 °C [7]. It is worth noting, that the viscosity values of both abovementioned INFs are still rather high and equal to 400 mPa·s at 25 °C (shear rate was not specified) [8] and 350 mPa·s at 25 °C and shear rate of 30 s^{-1} [7], respectively. Our previous study of INFs composed of commercial Nanocyl™ NC7000 MWCNTs with an aspect ratio of 150 has shown that the addition of 0.1 wt% of these nanoparticles into [C_2C_1im][SCN] (*i*) moderately increases the viscosity by 16.1% at 25 °C and shear rate of 32 s^{-1}, and (*ii*) negligibly enhances thermal conductivity by 1.7% at 25 °C [5]. We also found that the addition of 0.1 wt% in-house MWCNTs with an aspect ratio of 6300 into [C_2C_1im][SCN] negligibly reduces viscosity by 0.8% at 25 °C and shear rate of 32 s^{-1}, as well as nearly insignificantly enhances thermal conductivity by 1.7% at 25 °C [5]. However, it must be emphasized that the viscosities of both abovementioned cyano-based INFs are rather low and equal to 27.4 mPa·s and 23.4 mPa·s, respectively (both at 25 °C and shear rate of 32 s^{-1}) [5].

In this work, as a continuation of our previous study, we investigate the thermophysical properties of INFs composed of different weight concentrations (0.005%, 0.01%, 0.05%, 0.1%, 0.5%, and 1%) of carboxylic group-functionalized multi-walled carbon nanotubes (oMWCNTs), with an aspect ratio of 10,000, in 1-ethyl-3-methylimidazolium thiocyanate. The thermal conductivity and the density of pure IL and all INFs were measured in the temperature range from 25 °C to 50 °C. The viscosity of pristine IL and INFs was measured at 25 °C. The so-obtained characteristics prove the high performance of novel oMWCNT-based INFs as potential heat transfer media.

2. Materials and Methods

Materials. The 1-ethyl-3-methylimidazolium thiocyanate (0.98 mass fraction purity, chloride content ≤1 wt%) was purchased from IoLiTec (Heilbronn, Germany). The samples were dried and degassed under argon at 2 mbar (*Heidolph* rotary evaporator combined with the SC 920 G vacuum pump system) for 6 h and at 105 °C. The IL was constantly stored under argon. Water content determined by Karl Fischer method using TitroLine 7500 (SI Analytics, Mainz, Germany) was equal to 200 ppm. A brief specification of the [C_2C_1im][SCN] is presented in Table 1.

Table 1. Density, ρ, viscosity, η, and thermal conductivity, λ, of [C_2C_1im][SCN] at 25 °C in comparison with literature data.

	This Work	Literature
ρ (kg·m^{-3})	1116.1 ± 0.1	1115.20 [5], 1115.5 [9], 1115.9 [10,11], 1117 [12,13], 1122.26 [14]
η (mPa·s)	22.70 ± 3.9%	22.68 [15], 22.64 [5], 23.77 [16], 23.79 [17], 24.50 [12]
λ (W·m^{-1}·K^{-1})	0.179 ± 5%	0.177 [5], 0.184 [3]

The carboxyl-functionalized multi-walled carbon nanotubes (in-house 24 h oMWCNTs) were prepared using the 24-h catalytic chemical vapor deposition (c-CVD) process according to the previously reported protocol [5]. A brief specification of the in-house 24 h oMWCNTs is presented in Table 2. The c-CVD synthesized MWCNTs (in-house 24 h MWCNTs) (5.00 g) were introduced into a 250 mL round-bottomed flask. Subsequently, concentrated (96 wt%$_{aq}$) sulfuric (VI) acid (96 mL) and

concentrated (63 $wt\%_{aq}$) nitric (V) acid (32 mL) were added thereto. The reaction mixture was heated to boiling under the reflux condenser while a vigorous evolution of dark brown nitrogen (IV) oxide occurred. The boiling was continued for the next 10 min. The post-reaction mixture was left in the air, cooled for 15 min, and then poured into distilled water (3 L). Then the post-reaction mixture was filtered under reduced pressure through a G2 funnel. The product was collected on the funnel, washed with distilled water until neutral pH (15 L), and dried in an electric oven at 80 °C for 48 h. Yield was 3.21 g of brittled black solid.

Table 2. Characteristics of in-house 24 h oMWCNTs used in this study.

Name	Average Length (μm)	Average Diameter (nm)	Aspect Ratio (-)	Specific Surface Area ($m^2 \cdot g^{-1}$)	COOH Content ($mmol \cdot g^{-1}$)	OH Content ($mmol \cdot g^{-1}$)
in-house 24 h oMWCNTs	<500	~50	10,000	>22	1.0	0.4

Morphology of nanomaterials used in this study is shown in Figure 1 in the form of SEM images obtained by JSM-634OF FEG (JEOL, Akishima, Tokyo, Japan) at 5 kV and TEM micrographs acquired using 200 CX (JEOL, Akishima, Tokyo, Japan) at 200 kV.

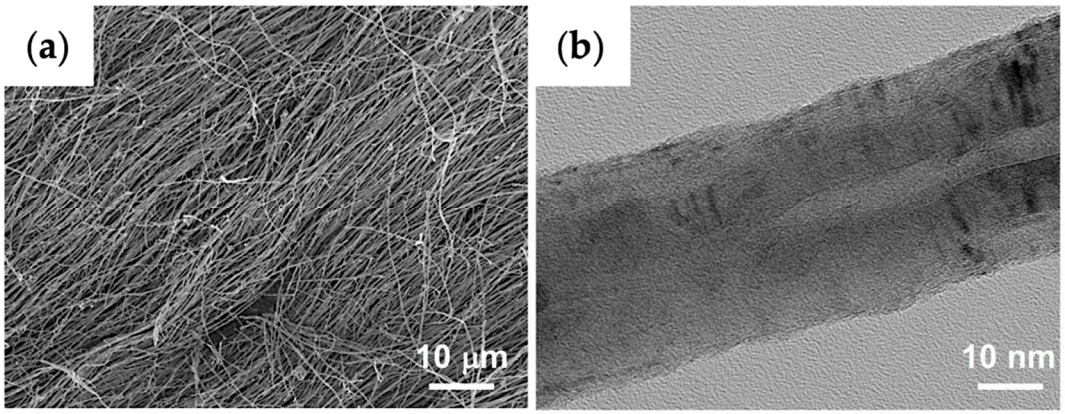
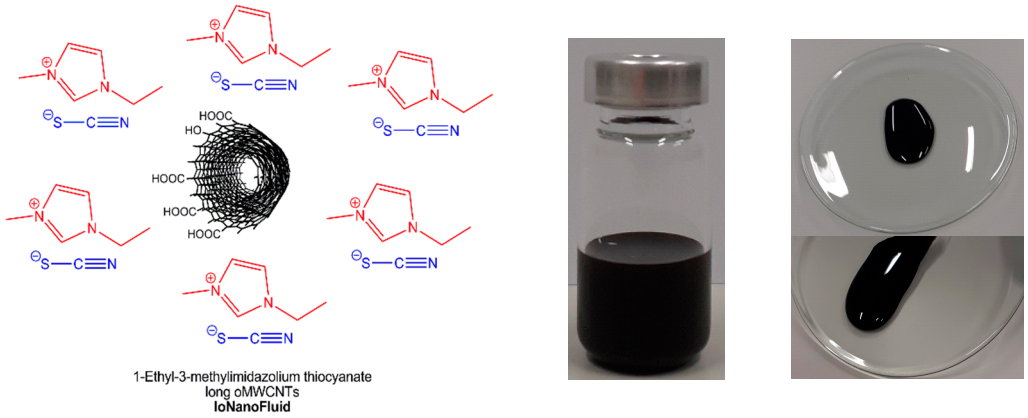


Figure 1. (**a**) Representative SEM image of oMWCNTs. (**b**) Representative TEM micrograph of oMWCNTs.

Sample preparation. The 20 mL of the samples of INFs (Figure 2) were prepared by dispersing various weight concentrations (0.005%, 0.01%, 0.05%, 0.1%, 0.5%, and 1%) of in-house 24 h oMWCNTs in base IL. The appropriate mass of the nanomaterial was determined using an analytical balance ACN220G (Axis, Gdańsk, Poland) with the accuracy of $\pm 1 \cdot 10^{-4}$ g. The obtained suspensions were sonicated for 10 min using probe sonicator UP200St (Hielscher Ultrasonics GmbH, Teltow, Germany) working at its nominal values of power, frequency, and amplitude (200 W, 26 kHz, and 100%, respectively). During ultrasound treatment, the samples were cooled by using a jacketed beaker with tap water as the working liquid.

Thermal conductivity measurements. The thermal conductivity was measured via the hot-wire technique using KD2 Pro Thermal Properties Analyzer (Decagon Devices Inc., Pullman, WA, USA) with KS-1 sensor. The measurement uncertainty was ±5% (at 95% confidence level). Additionally, the KD2 Pro analyzer was precalibrated using a supplied verification standard of glycerin. All samples were thermostated at 25 °C, 30 °C, 35 °C, 40 °C, 45 °C, and 50 °C by Open Bath Circulator ED-5 (Julabo GmbH, Seelbach, Germany) with ethylene glycol as a working liquid for a minimum of 30 min until thermal equilibrium was obtained. Three readings of the KD2 Pro analyzer with a correlation value of $R^2 \geq 0.9999$ were recorded at the 15-minute interval and then averaged to get the thermal conductivity of each sample at each temperature.



(**a**) (**b**)

Figure 2. (**a**) Chemical structure of in-house 24 h oMWCNT/[C_2C_1im][SCN]. (**b**) The pictures of IoNanofluids (INF) composed of 1 wt% in-house 24 h oMWCNTs + [C_2C_1im][SCN].

Viscosity measurements. The apparent viscosity was determined using rotary viscometer LV DV-II+Pro (Brookfield Engineering, Middleboro, MA, USA) with DIN-86 and DIN-87 spindles. The measurements were carried out at a constant temperature of 25 ± 0.1 °C which was carefully controlled by a resistance temperature detector with an expanded uncertainty of ±1 °C and a resolution of 0.1 °C. The appropriate temperature was provided by Open Bath Circulator ED-5 (Julabo GmbH, Seelbach, Germany) with ethylene glycol as a working liquid. Each sample was stabilized at the given temperature for a minimum of 30 min until thermal equilibrium was reached. The expanded relative uncertainty (k = 2) of viscosity measurements was equal to ±3.9% and ±5.4% for spindles DIN-86 and DIN-87, respectively.

Density measurements. The density of pure IL and INFs was measured via vibrating-tube densimeters DMA 5000M and DMA 5000 (Anton Paar, Graz, Austria), respectively. The devices were calibrated with dry air and high-quality Millipore water, density certified, with electrolytic conductivity of 1×10^{-4} S·m^{-1} at 25 °C. In order to achieve the minimum possible uncertainty over the whole temperature range studied, the calibration in the temperature range under investigations has been conducted. The viscosity corrections were automatically made. Expanded density uncertainties were equal to ±0.1 kg·m^{-3} and ±0.3 kg·m^{-3} for measurements of pure IL and INFs, respectively. The temperature was measured with an expanded uncertainty of ±0.02 °C.

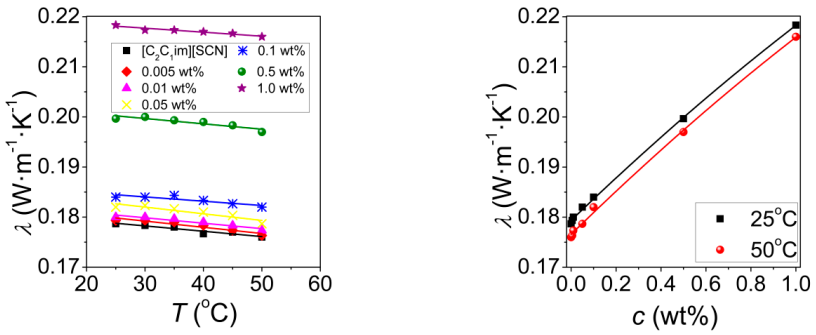
3. Results

3.1. Thermal Conductivity

The thermal conductivity of each sample was measured three times in the temperature range from 25 °C to 50 °C in 5 °C steps. The experimental values are listed in Table 3 and presented in Figure 3. The thermal conductivity of pristine [C_2C_1im][SCN] obtained in this work at 25 °C is in an excellent agreement with the experimental data reported by França et al. [3] and previously reported by us [5], i.e., the relative deviations, *RD*s ($RD/\% = 100 \cdot (\lambda_{\text{this work}} - \lambda_{\text{lit}})/\lambda_{\text{this work}}$) are lower than declared measurement uncertainty and equal to −2.8% and 1.1%, respectively.

Table 3. Thermal conductivity of ionic liquids (IL) and INFs in the temperature range from 25 °C to 50 °C.

T (°C)	λ (W·m^{-1}·K^{-1})							
	Series 1	**Series 2**	**Series 3**	**Mean**	**Series 1**	**Series 2**	**Series 3**	**Mean**
	[C_2C_1im][SCN]				0.1 wt% in-house 24 h oMWCNTs + [C_2C_1im][SCN]			
25	0.178	0.179	0.179	0.179	0.184	0.184	0.184	0.184
30	0.178	0.178	0.179	0.178	0.184	0.184	0.184	0.184
35	0.178	0.178	0.178	0.178	0.184	0.185	0.184	0.184
40	0.177	0.177	0.176	0.177	0.183	0.183	0.184	0.183
45	0.177	0.177	0.177	0.177	0.182	0.183	0.183	0.183
50	0.176	0.176	0.176	0.176	0.182	0.181	0.181	0.181
	0.005 wt% in-house 24 h oMWCNTs + [C_2C_1im][SCN]				0.5 wt% in-house 24 h oMWCNTs + [C_2C_1im][SCN]			
25	0.179	0.180	0.179	0.179	0.200	0.199	0.200	0.200
30	0.179	0.179	0.180	0.179	0.200	0.200	0.200	0.200
35	0.179	0.179	0.179	0.179	0.199	0.199	0.200	0.199
40	0.179	0.178	0.178	0.178	0.199	0.199	0.199	0.199
45	0.177	0.178	0.177	0.177	0.199	0.199	0.197	0.198
50	0.176	0.176	0.177	0.176	0.198	0.196	0.197	0.197
	0.01 wt% in-house 24 h oMWCNTs + [C_2C_1im][SCN]				1 wt% in-house 24 h oMWCNTs + [C_2C_1im][SCN]			
25	0.180	0.180	0.180	0.180	0.220	0.217	0.218	0.218
30	0.180	0.180	0.180	0.180	0.217	0.218	0.217	0.217
35	0.180	0.180	0.179	0.180	0.217	0.217	0.218	0.217
40	0.179	0.179	0.179	0.179	0.217	0.217	0.217	0.217
45	0.179	0.178	0.178	0.178	0.216	0.217	0.217	0.217
50	0.178	0.178	0.176	0.177	0.216	0.217	0.215	0.216
	0.05 wt% in-house 24 h oMWCNTs + [C_2C_1im][SCN]							
25	0.182	0.182	0.182	0.182				
30	0.183	0.182	0.182	0.182				
35	0.182	0.181	0.182	0.182				
40	0.181	0.181	0.181	0.181				
45	0.181	0.180	0.180	0.180				
50	0.179	0.179	0.178	0.179				

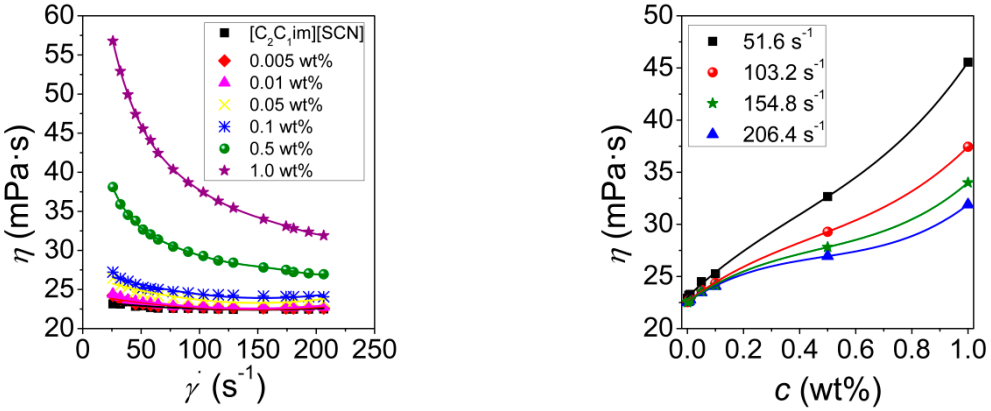


(**a**) (**b**)

Figure 3. (**a**) The influence of temperature on the thermal conductivity of IL and INFs. (**b**) Thermal conductivity of INFs at 25 °C and 50 °C as a function of weight concentration, c, of in-house 24 h oMWCNTs.

3.2. Viscosity

The viscosity of the IL and INFs under test was measured at 25 °C within the range of shear rate from 25.8 s^{-1} to 206.4 s^{-1}. The results are presented in Figure 4. Pure [C_2C_1im][SCN] turned out to be a Newtonian liquid with constant viscosity of 22.70 mPa·s ± 3.9%, which is in good agreement with the literature data reported by Vataščin et al. [15], Wang et al. [16], Larriba et al. [17], Freire et al. [12], and previously reported by us [5], i.e., the relative deviations, *RD*s ($RD/\% = 100\cdot(\eta_{\text{this work}} - \eta_{\text{lit}})/\eta_{\text{this work}}$) are equal to 0.04%, −4.7%, −4.8%, −7.9%, and 0.26%, respectively.

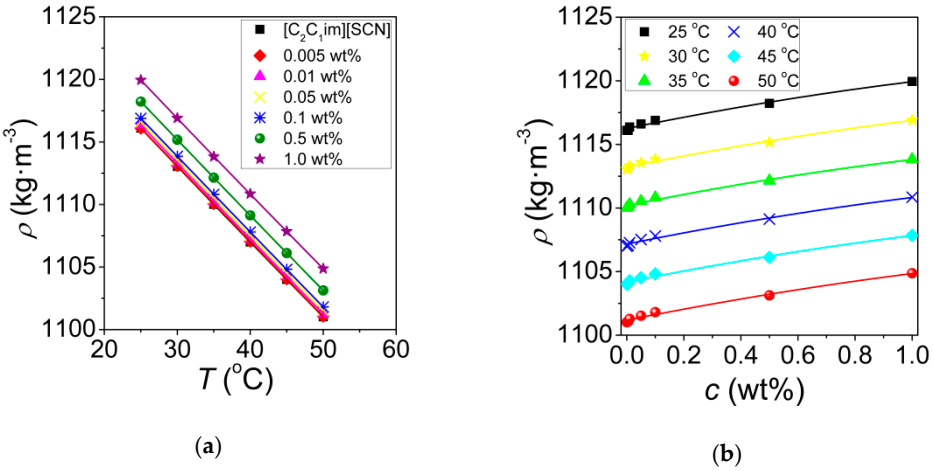


(**a**) (**b**)

Figure 4. (**a**) The viscosity curves of IL and INFs at 25 °C. (**b**) The viscosity of IL and INFs at 25 °C as a function of weight concentration of in-house 24 h oMWCNTs, c, for a specified shear rate of 51.6 s^{-1}, 103.2 s^{-1}, 154.8 s^{-1}, and 206.4 s^{-1}.

3.3. Density

The density of the IL and INFs was measured within the temperature range from 25 °C to 50 °C. The experimental values are collected in Table 4 and presented in Figure 5. The density of pristine [C_2C_1im][SCN] obtained in this work at 25 °C is in a very good agreement with the literature data [5,9–14] (Table 1). The relative deviations, *RD*s ($RD/\% = 100\cdot(\rho_{\text{this work}} - \rho_{\text{lit}})/\rho_{\text{this work}}$), are equal to −0.55% [14], −0.08% [12,13], 0.02% [10,11], 0.05% [9], and 0.08% for result reported recently by us [5].



(**a**) (**b**)

Figure 5. (**a**) The influence of temperature on the density of IL and INFs. (**b**) The density of INFs as a function of weight concentration, c, of in-house 24 h oMWCNTs.

Table 4. Density, ρ, of IL and INFs in the temperature range from 25 °C to 50 °C.

T (°C)	ρ (kg·m^{-3})	T (°C)	ρ (kg·m^{-3})	T (°C)	ρ (kg·m^{-3})
	[C_2C_1im][SCN]		0.01 wt% in-house 24 h oMWCNTs + [C_2C_1im][SCN]		0.1 wt% in-house 24 h oMWCNTs + [C_2C_1im][SCN]
25	1116.10	25	1116.37	25	1116.89
30	1113.05	30	1113.33	30	1113.85
35	1110.02	35	1110.29	35	1110.82
40	1107.00	40	1107.27	40	1107.80
45	1104.01	45	1104.27	45	1104.82
50	1101.02	50	1101.28	50	1101.81
	0.005 wt% in-house 24 h oMWCNTs + [C_2C_1im][SCN]		0.05 wt% in-house 24 h oMWCNTs + [C_2C_1im][SCN]		0.5 wt% in-house 24 h oMWCNTs + [C_2C_1im][SCN]
25	1116.11	25	1116.61	25	1118.23
30	1113.12	30	1113.57	30	1115.18
35	1110.08	35	1110.54	35	1112.15
40	1107.06	40	1107.52	40	1109.13
45	1104.06	45	1104.52	45	1106.12
50	1101.07	50	1101.53	50	1103.13
	1 wt% in-house 24 h oMWCNTs + [C_2C_1im][SCN]				
25	1119.95				
30	1116.91				
35	1113.83				
40	1110.85				
45	1107.85				
50	1104.87				

4. Discussion and Conclusions

The thermal conductivity of IL and INFs slightly decreases with temperature and almost linearly increases with increasing weight concentration of in-house 24 h oMWCNTs (Figure 3). The highest 22% enhancement in the thermal conductivity of INFs was observed at 1 wt% loading of nanoparticles. The thermal conductivity increase for INFs composed of [C_2C_1im][SCN] and 0.5 wt% or 1 wt% carbon nanotubes depends on their morphology as follows: (Nanocyl^{TM} NC7000 MWCNTs [5] = Baytubes^{TM} C150 HP MWCNTs [3]) < in-house 24 h oMWCNTs < in-house 16 h MWCNTs [5]. The Nanocyl^{TM} NC7000 MWCNTs are characterized by only slightly smaller outer diameter (9.5 nm) and length (1.5 μm) than Baytubes™ C150 HP MWCNTs (13–16 nm and 1–10 μm, respectively). Strikingly different are in-house 24 h oMWCNTs revealing entangled fibrous morphologies (d ~ 50 nm, l < 500 μm) maintained by a dense three-dimensional network of hydrogen bonding between carboxyl (and hydroxyl) groups and van der Waals forces. The COOH content in in-house 24 h oMWCNTs, as revealed by Boehm titration, was equal to 1.0 mmol g^{-1} (and it was accompanied by 0.4 mmol g^{-1} of hydroxyl -OH groups). Such a structure leads to the enhanced, prolonged stability of INFs since apart from π-cation interactions between the outer nanotube walls and IL cations, also CNT-(CO)O-H—cation interactions are possible. The INFs with low loading of in-house 24 h oMWCNTs (≤0.01 wt%) exhibit approximately Newtonian behavior. The higher concentrations of nanoparticles (>0.01 wt%) cause a formation of larger carbon agglomerates in IL, which increase flow resistance and non-Newtonian shear thinning (pseudoplastic) behavior (Figure 4).

Recently, we found that the addition of 0.1 wt% in-house 7 h MWCNTs with an aspect ratio of 6300 into [C_2C_1im][SCN] negligibly reduces the viscosity by 0.8% at 25 °C and shear rate of 32 s^{-1}, as well as negligibly enhances the thermal conductivity by 1.7% at 25 °C [5]. In this work, the loading of 0.1 wt%

in-house 24 h oMWCNTs negligibly increases the viscosity by 3.9% at 25 °C and shear rate of 32 s^{-1}, as well as negligibly enhances the thermal conductivity by 0.37% at 25 °C. Much more significant changes can be seen under identical conditions in the case of INF composed of 1 wt% in-house 7 h MWCNTs and $[C_2C_1im][SCN]$, i.e., moderate viscosity of 135 mPa·s and remarkably high thermal conductivity enhancement of 43.1% [5]. In turn, the INF composed of 1 wt% in-house 24 h oMWCNTs under similar conditions exhibits a much lower viscosity of 52.92 mPa·s and a less spectacular increase in thermal conductivity of 22%. Nevertheless, the viscosity of 1 wt% nanodispersion studied in this work is much closer to the pristine ILs, and importantly, the thermal conductivity of the sample is still significant.

Thus, the INFs composed of in-house 24 h oMWCNTs and $[C_2C_1im][SCN]$ can become potential candidates for heat transfer media with the optimized heat transfer capability (resulting from thermal conductivity) and internal resistance (resulting from viscosity).

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